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Synthesis and Structures of Scandium and Lutetium Benzyl Complexes

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Supporting Information

Table S-1. Crystal data and structure refinement for **1**.

Empirical formula	C33 H45 O3 Sc
Formula weight	534.67
Temperature	100(1) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, C c
Unit cell dimensions	a = 17.202(4) A alpha = 90 deg. b = 20.759(5) A beta = 93.695(4) deg. c = 8.267(2) A gamma = 90 deg.
Volume	2946.0(12) A^3
Z, Calculated density	4, 1.205 Mg/m^3
Absorption coefficient	0.280 mm^-1
F(000)	1152
Crystal size	0.53 x 0.16 x 0.10 mm
Theta range for data collection	2.84 to 27.10 deg.
Limiting indices	-22<=h<=21, -26<=k<=26, -10<=l<=10
Reflections collected / unique	12243 / 5994 [R(int) = 0.0387]
Completeness to theta = 25.00	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9724 and 0.8519
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5994 / 2 / 514
Goodness-of-fit on F^2	1.009
Final R indices [I>2sigma(I)]	R1 = 0.0399, wR2 = 0.0824
R indices (all data)	R1 = 0.0471, wR2 = 0.0852
Absolute structure parameter	0.03(2)
Largest diff. peak and hole	0.228 and -0.234 e.A^-3

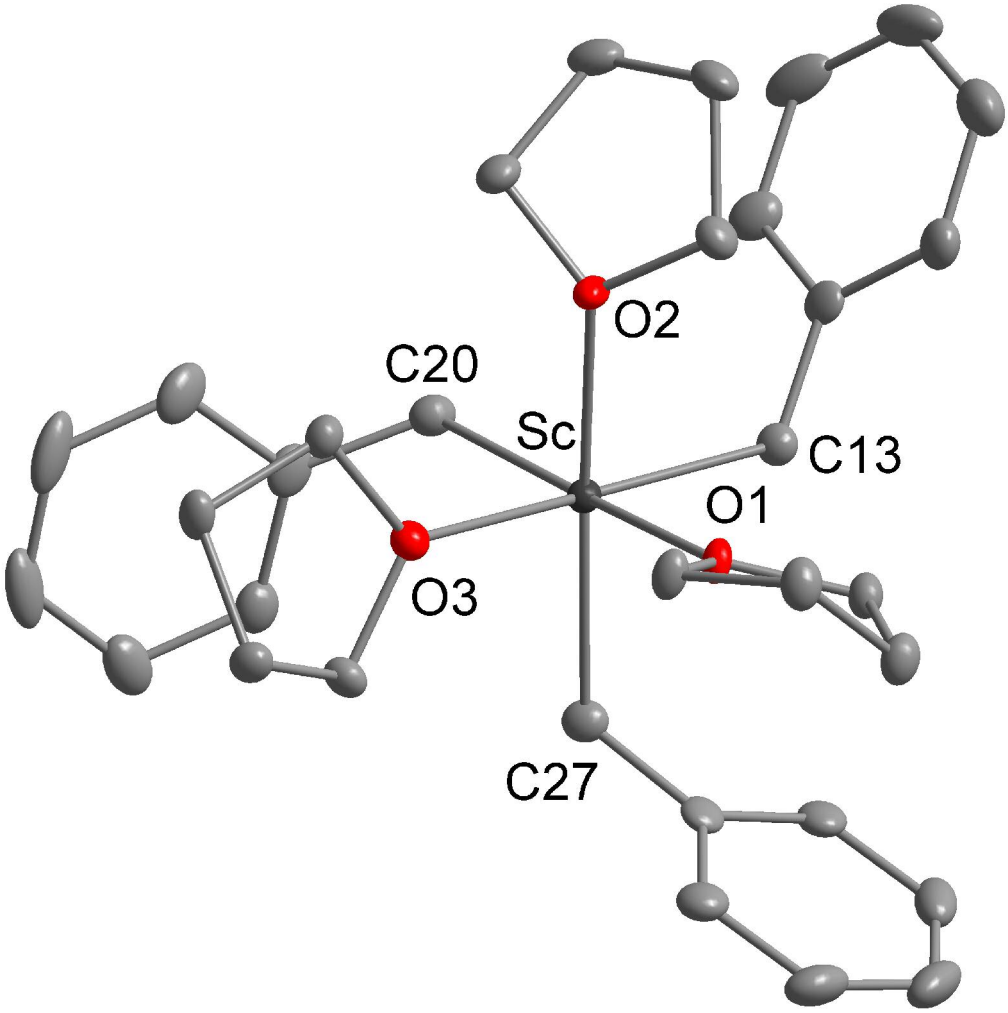


Figure S-1. Molecular structure of **1**, with 50% probability ellipsoids. Hydrogen atoms are omitted for clarity.

Table S-2. Crystal data and structure refinement for **2**.

Empirical formula	C29 H37 O2 Sc
Formula weight	462.57
Temperature	100(1) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P -1
Unit cell dimensions	a = 7.6779(6) A alpha = 86.423(1) deg. b = 11.3723(9) A beta = 82.664(1) deg. c = 14.824(1) A gamma = 78.332(1) deg.
Volume	1256.35(16) A^3
Z, Calculated density	2, 1.223 Mg/m^3
Absorption coefficient	0.315 mm^-1
F(000)	496
Crystal size	0.47 x 0.27 x 0.21 mm
Theta range for data collection	2.73 to 28.28 deg.
Limiting indices	-10<=h<=10, -15<=k<=14, -19<=l<=18
Reflections collected / unique	11481 / 5968 [R(int) = 0.0164]
Completeness to theta = 25.00	98.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9359 and 0.8622
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5968 / 0 / 437
Goodness-of-fit on F^2	1.040
Final R indices [I>2sigma(I)]	R1 = 0.0418, wR2 = 0.1043
R indices (all data)	R1 = 0.0502, wR2 = 0.1105
Absolute structure parameter	?
Largest diff. peak and hole	0.732 and -0.458 e.A^-3

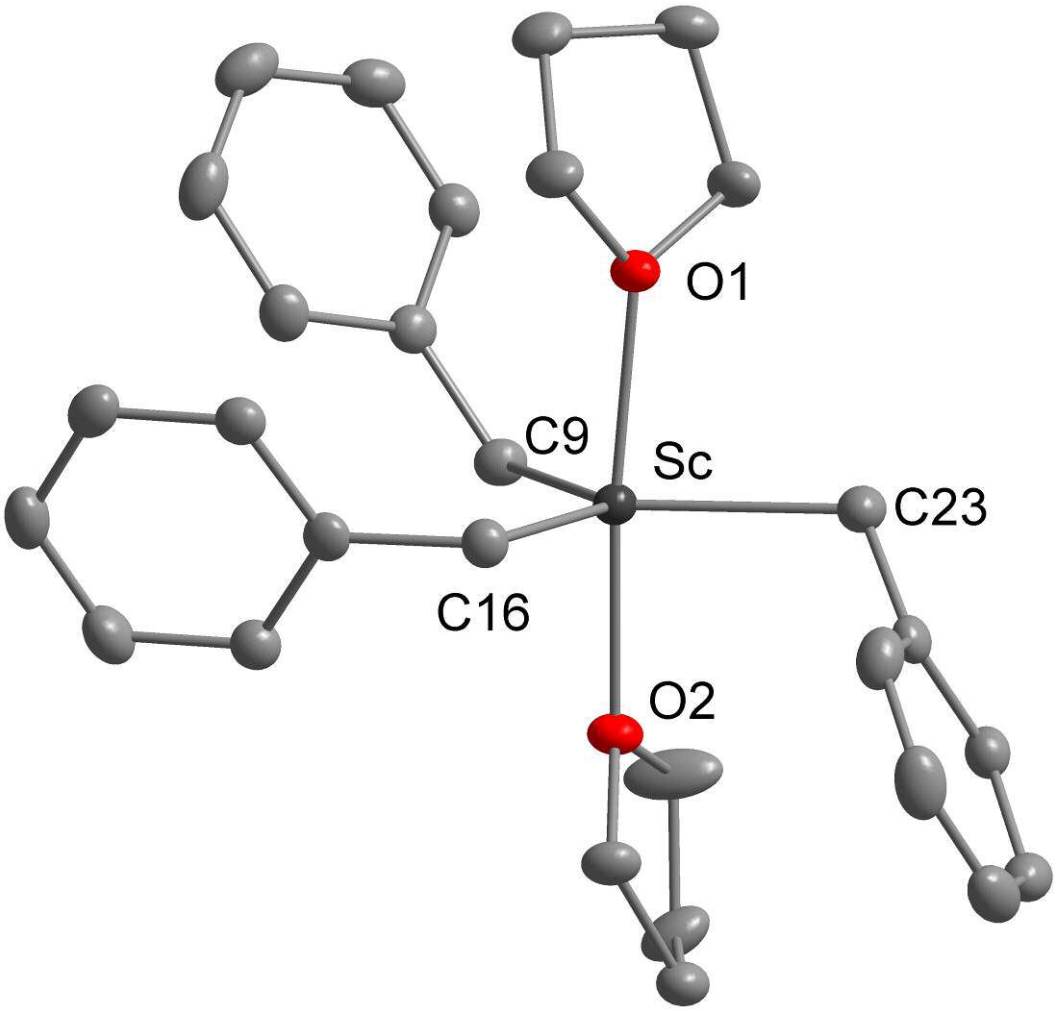


Figure S-2. Molecular structure of **1**, with 50% probability ellipsoids. Hydrogen atoms are omitted for clarity.

Table S-3. Crystal data and structure refinement for **3**.

Empirical formula	C47 H59 K2 O3 Sc
Formula weight	795.10
Temperature	200(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pna2(1)
Unit cell dimensions	a = 25.471(5) A alpha = 90 deg. b = 9.2954(19) A beta = 90 deg. c = 18.336(4) A gamma = 90 deg.
Volume	4341.3(15) A^3
Z, Calculated density	4, 1.216 Mg/m^3
Absorption coefficient	0.399 mm^-1
F(000)	1696
Crystal size	0.5 x 0.15 x 0.1 mm
Theta range for data collection	2.93 to 29.25 deg.
Limiting indices	-28<=h<=34, -12<=k<=11, -21<=l<=25
Reflections collected / unique	19529 / 10588 [R(int) = 0.0493]
Completeness to theta = 29.25	98.4 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	10588 / 1 / 446
Goodness-of-fit on F^2	0.939
Final R indices [I>2sigma(I)]	R1 = 0.0568, wR2 = 0.1269
R indices (all data)	R1 = 0.1011, wR2 = 0.1427
Absolute structure parameter	0.01(4)
Largest diff. peak and hole	1.002 and -0.433 e.A^-3

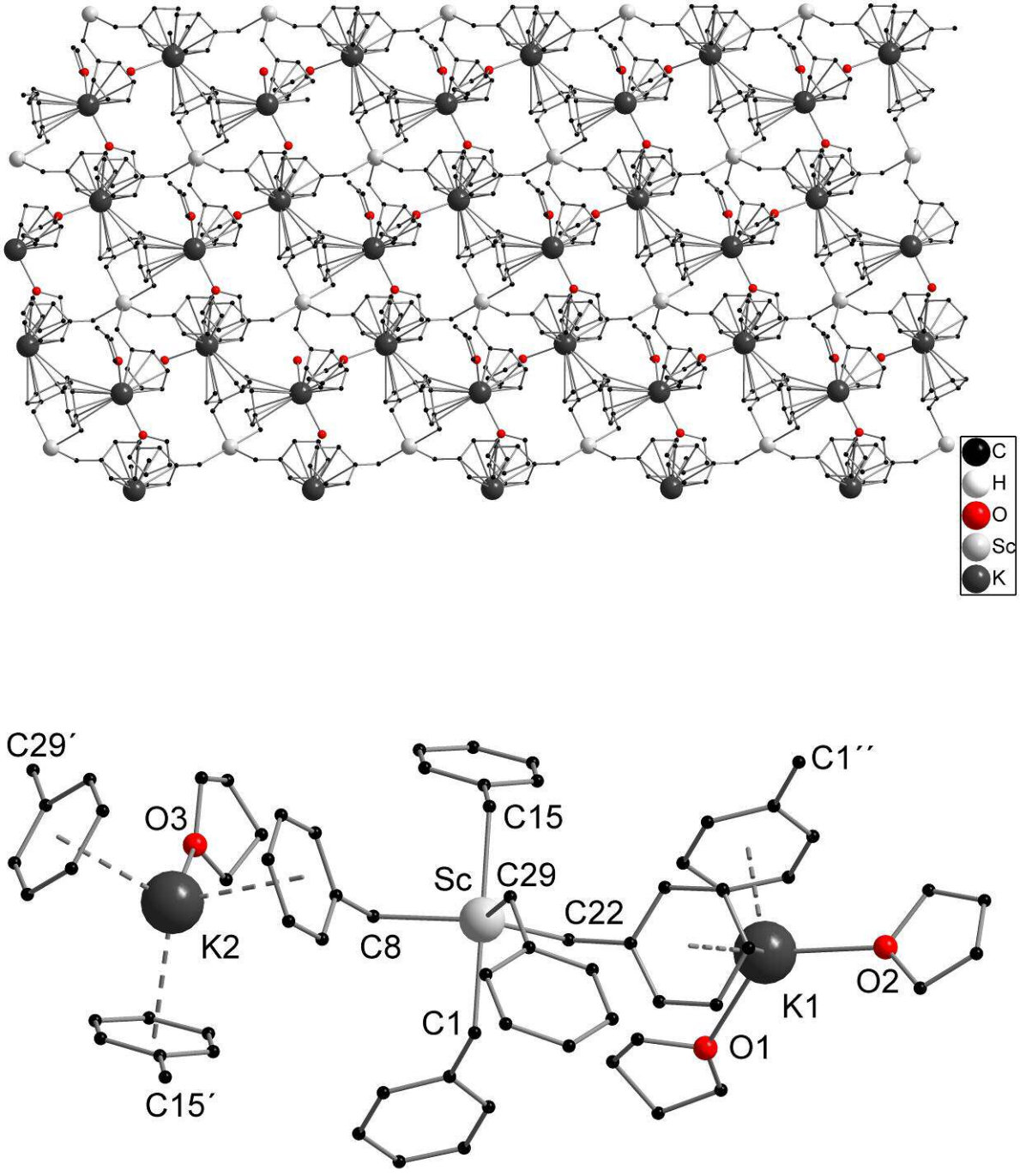


Figure S-3. Solid-state structure of **3**. Top: View to a single sheet from above. Bottom: Shown is one unit out of the polymeric structure and the atom labeling scheme, omitting hydrogen atoms.

Table S-4. Crystal data and structure refinement for **4**.

Identification code	z:\daten\nils\nils52b\nils52b
Empirical formula	C33 H45 Lu O3
Formula weight	664.66
Temperature	200(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, C c
Unit cell dimensions	a = 17.3716(9) A alpha = 90 deg. b = 20.9550(14) A beta = 93.571(4) deg. c = 8.3958(4) A gamma = 90 deg.
Volume	3050.3(3) A^3
Z, Calculated density	4, 1.447 Mg/m^3
Absorption coefficient	3.266 mm^-1
F(000)	1352
Crystal size	0.458 x 0.240 x 0.079 mm
Theta range for data collection	2.81 to 25.02 deg.
Limiting indices	-20<=h<=20, -24<=k<=24, -9<=l<=9
Reflections collected / unique	13424 / 5033 [R(int) = 0.0381]
Completeness to theta = 25.02	99.5 %
Absorption correction	Integration
Max. and min. transmission	0.7403 and 0.4640
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5033 / 2 / 334
Goodness-of-fit on F^2	0.985
Final R indices [I>2sigma(I)]	R1 = 0.0203, wR2 = 0.0449
R indices (all data)	R1 = 0.0228, wR2 = 0.0454
Absolute structure parameter	-0.001(8)
Largest diff. peak and hole	0.395 and -0.669 e.A^-3

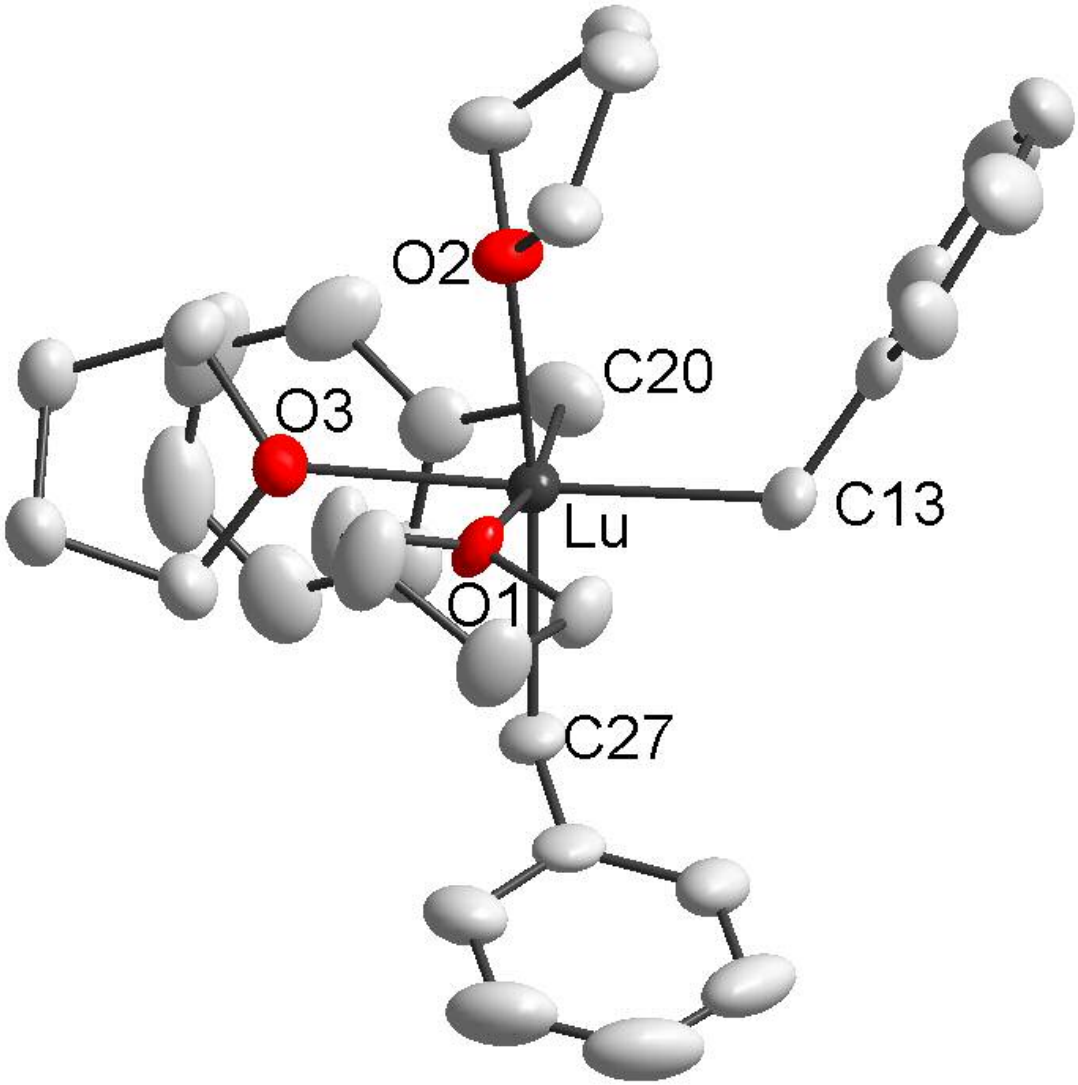


Figure S-4. Molecular structure of **4**, with 50% probability ellipsoids. Hydrogen atoms are omitted for clarity.

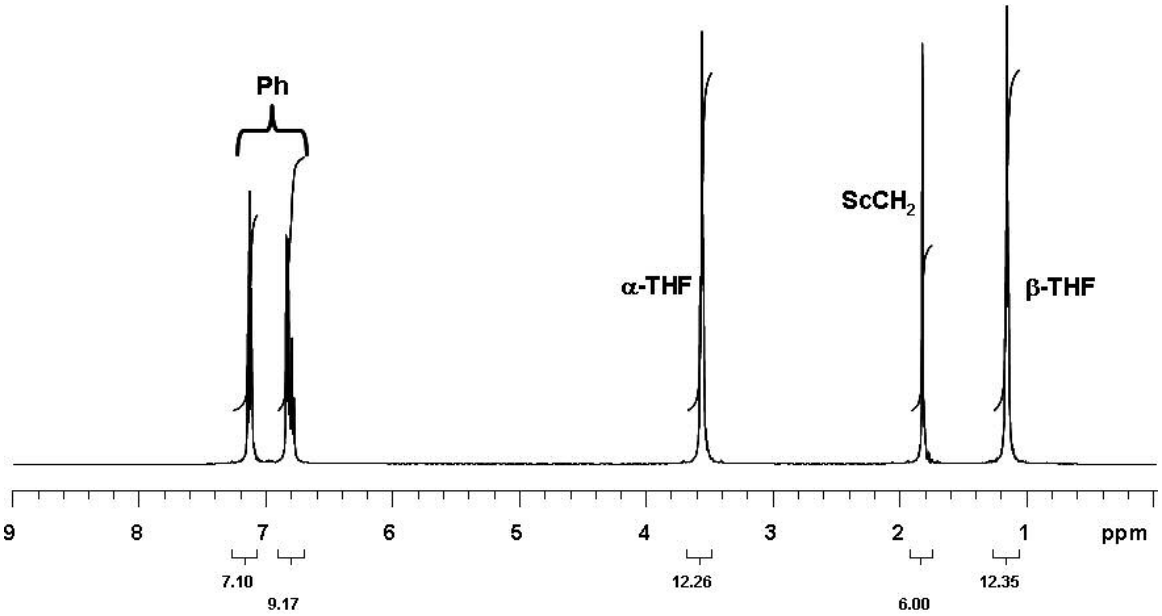


Figure S-5. ^{1}H NMR spectrum (500 MHz, C_6D_6, 20°C) of $Sc(CH_2Ph)_3(THF)_3$ (**1**).

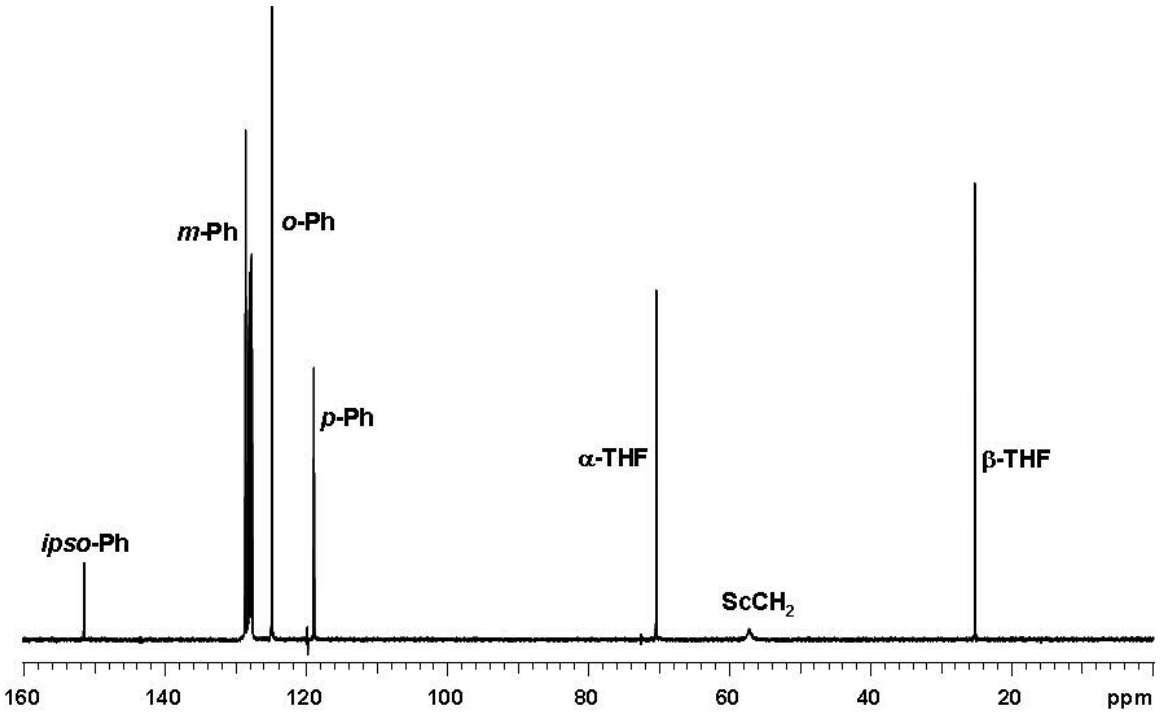


Figure S-6. ^{13}C{H} NMR spectrum (125.7 MHz, C_6D_6, 20°C) of $Sc(CH_2Ph)_3(THF)_3$ (**1**).

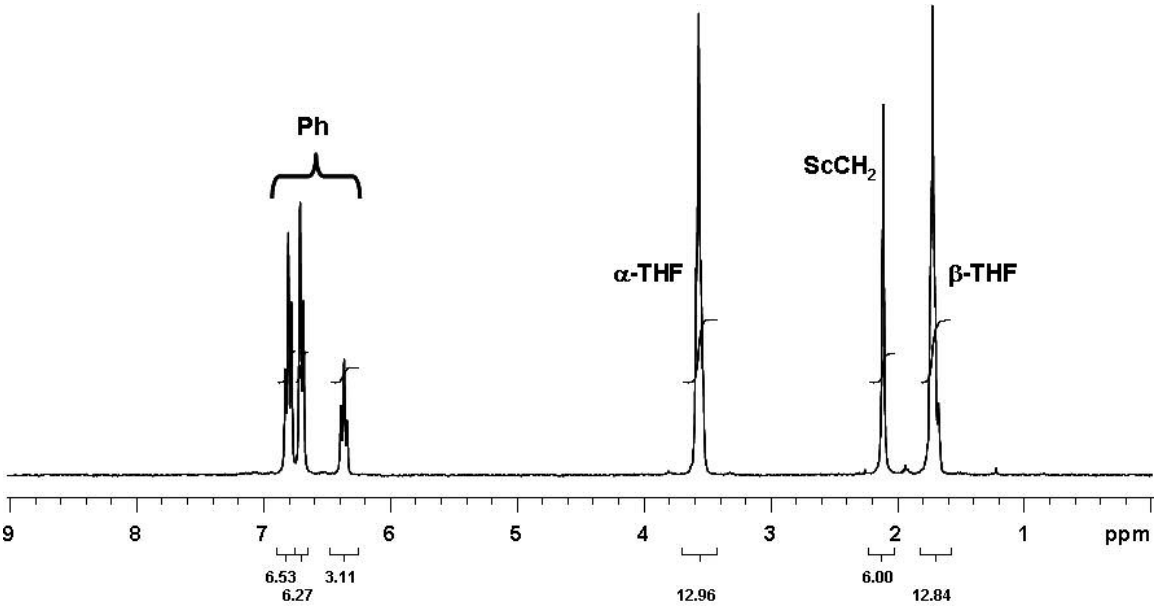


Figure S-7. ^{1}H NMR spectrum (500 MHz, THF-d_8, 20°C) of $Sc(CH_2Ph)_3(THF)_3$ (**1**).

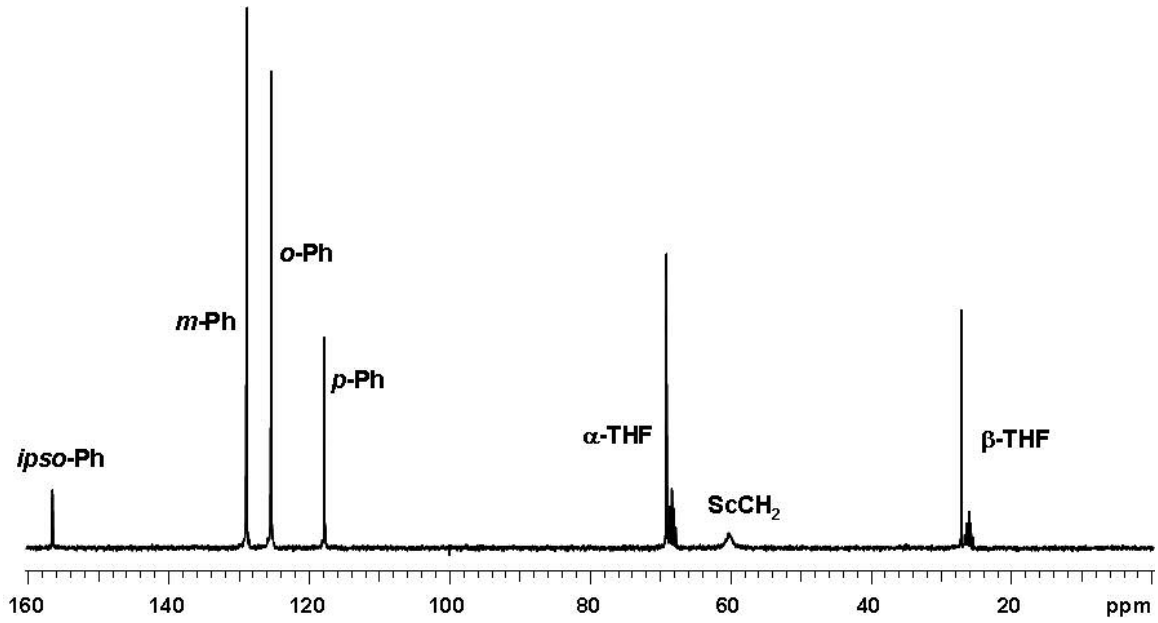


Figure S-8. ^{13}C{H} NMR spectrum (125.7 MHz, THF-d_8, 20°C) of $Sc(CH_2Ph)_3(THF)_3$ (**1**).

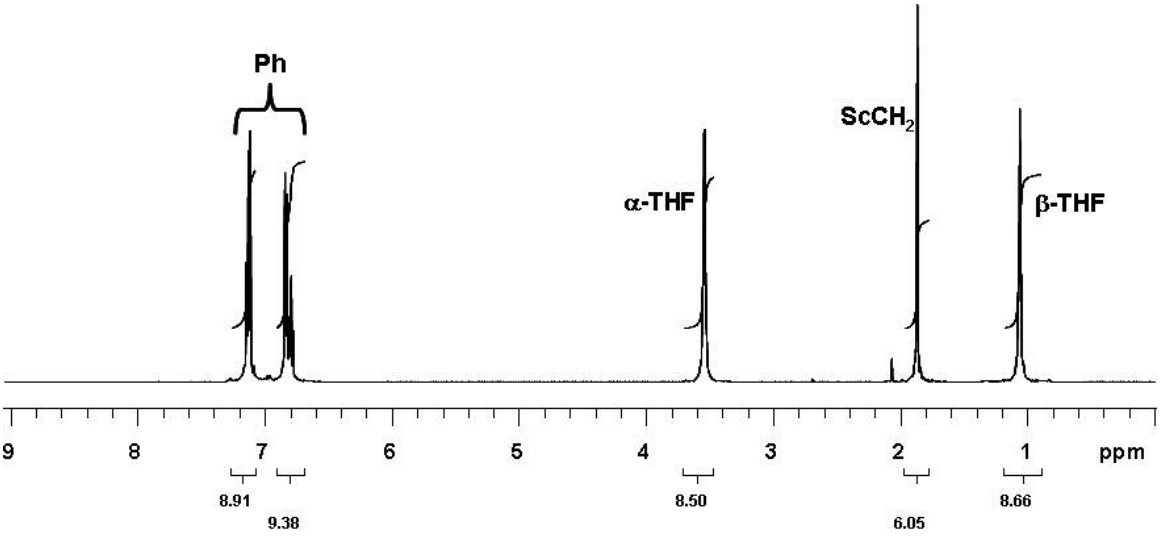


Figure S-9. ^{1}H NMR spectrum (500 MHz, C_6D_6, 20°C) of $Sc(CH_2Ph)_3(THF)_2$ (**2**).

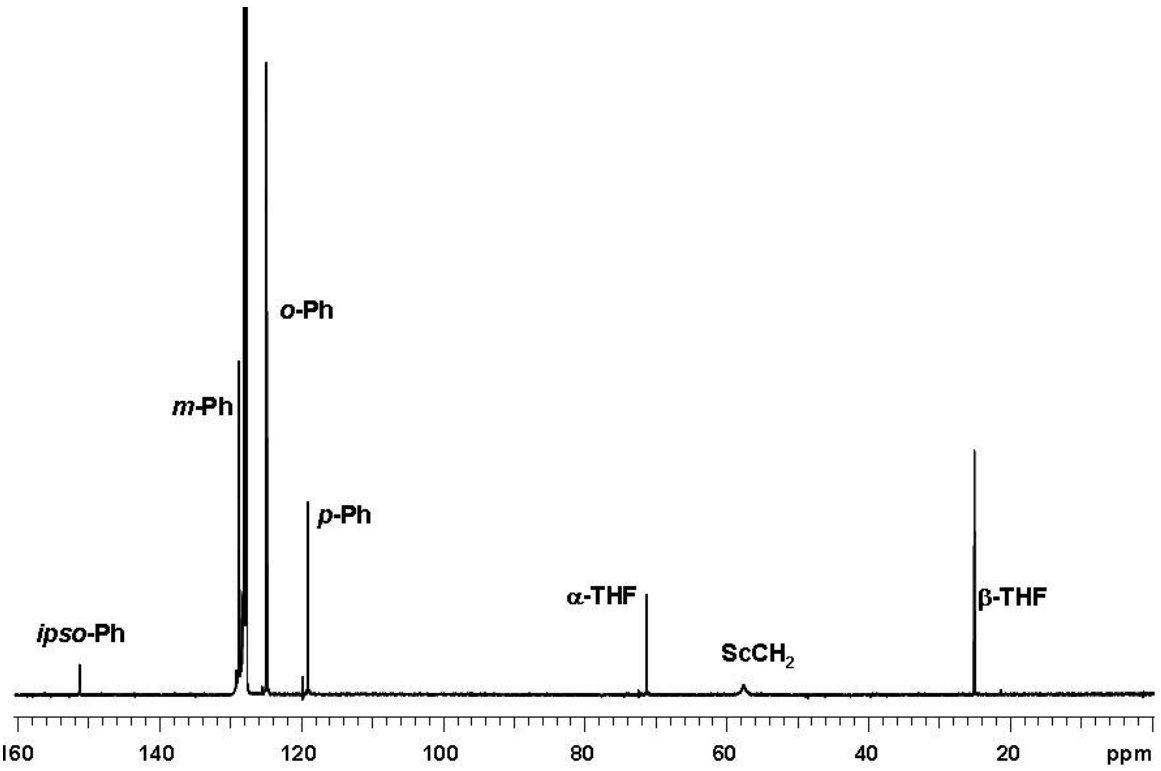


Figure S-10. ^{13}C{H} NMR spectrum (125.7 MHz, C_6D_6, 20°C) of $Sc(CH_2Ph)_3(THF)_2$ (**2**).

Figure S-11. ^{1}H NMR spectrum of **3**

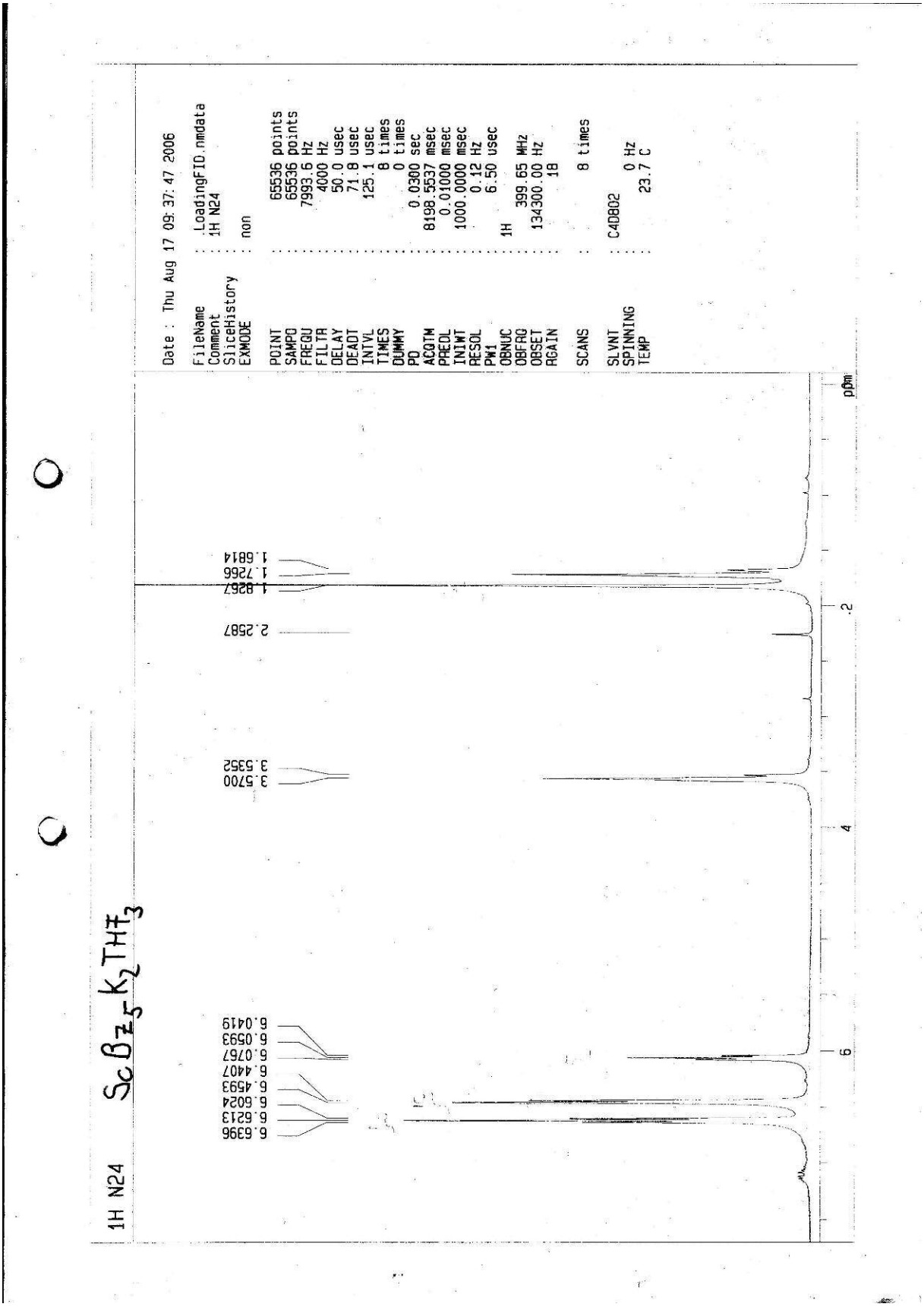
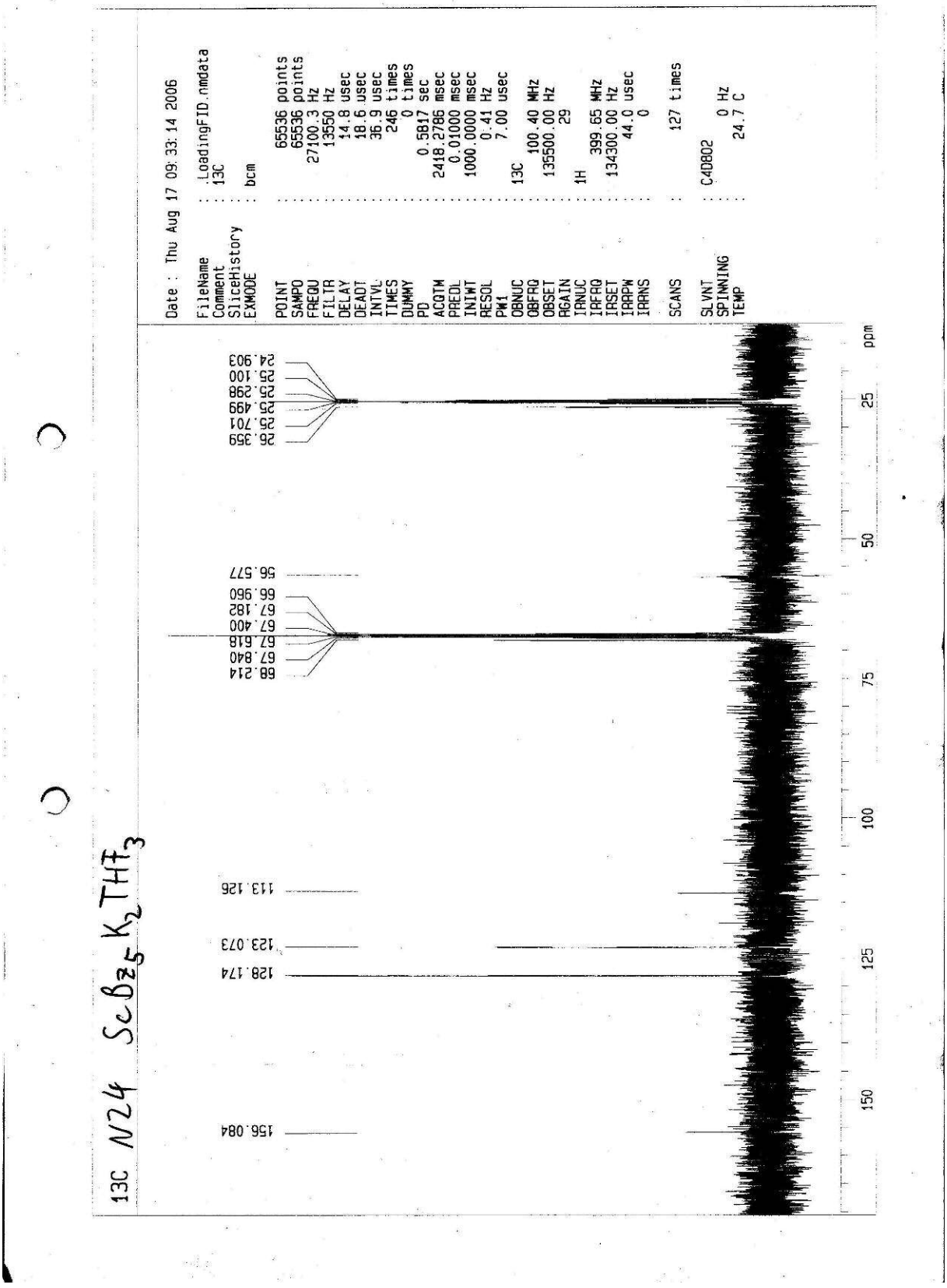


Figure S-12. ^{13}C NMR spectrum of **3**



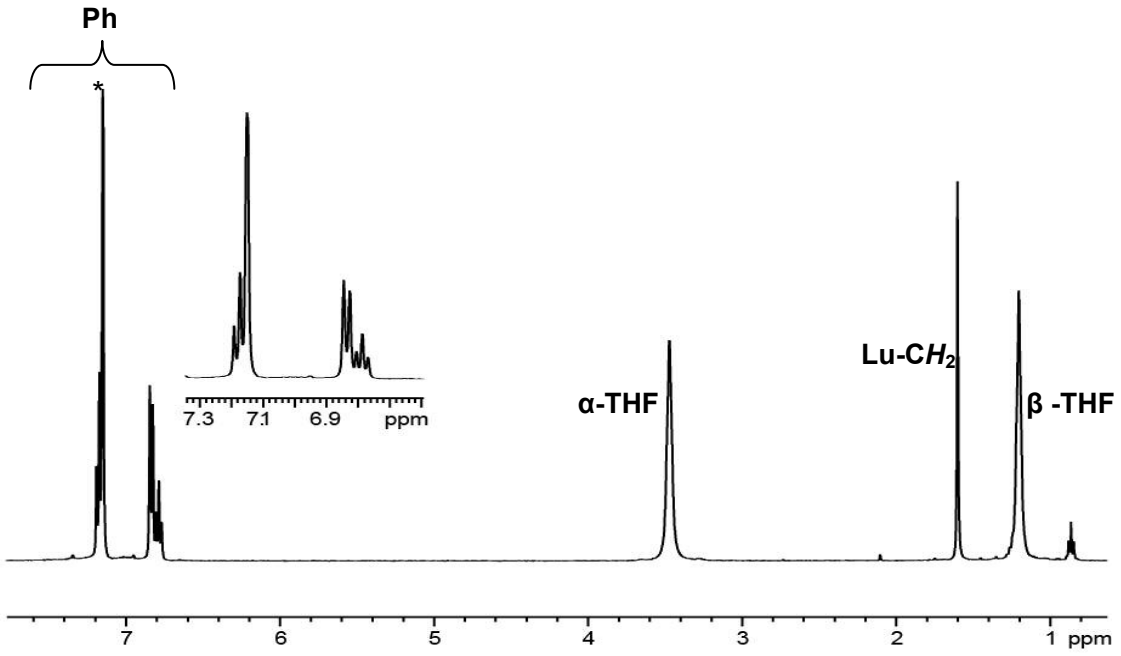


Figure S-13. ^{1}H NMR spectrum (400 MHz, C_6D_6, 27°C) of $Lu(CH_2Ph)_3(THF)_3$ (**4**).

* denotes residual solvent signal.

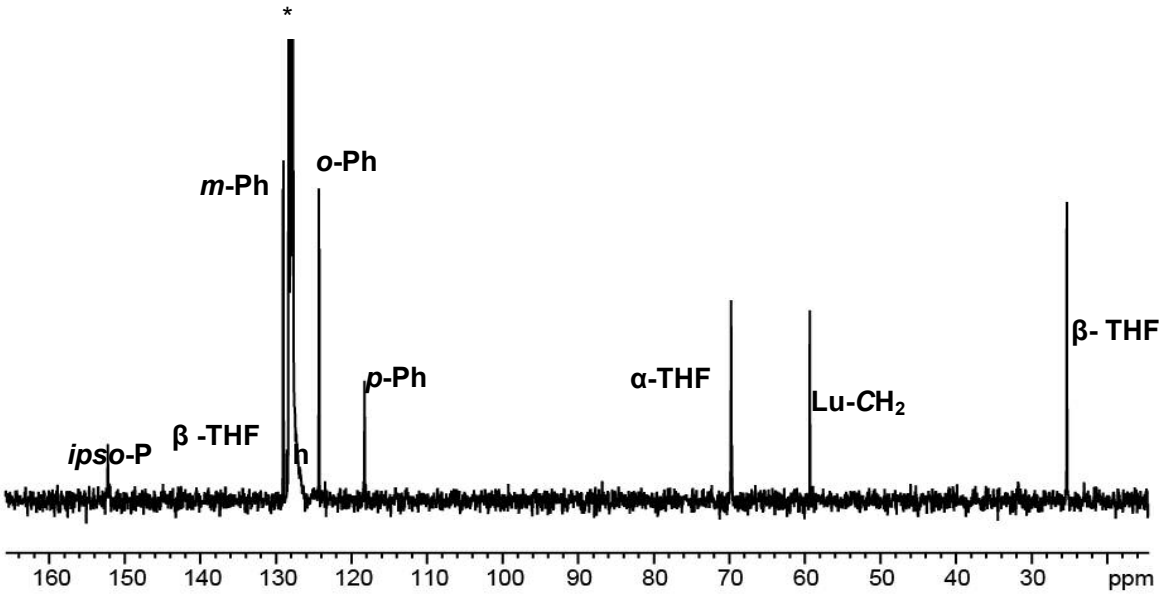


Figure S-14. ^{13}C NMR spectrum (100.58 MHz, C_6D_6, 27°C) of $Lu(CH_2Ph)_3(THF)_3$ (**4**).

* denotes residual solvent signal.

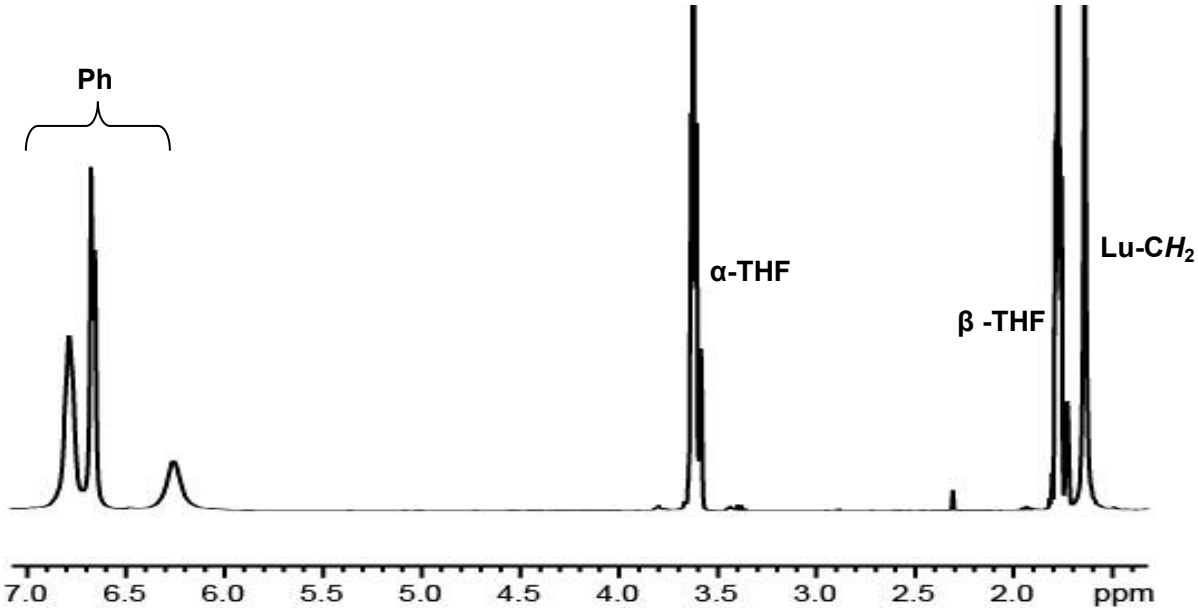


Figure S-15. ^{1}H NMR spectrum (400 MHz, THF-d_8, 27°C) of $Lu(CH_2Ph)_3(THF)_3$ (**4**).

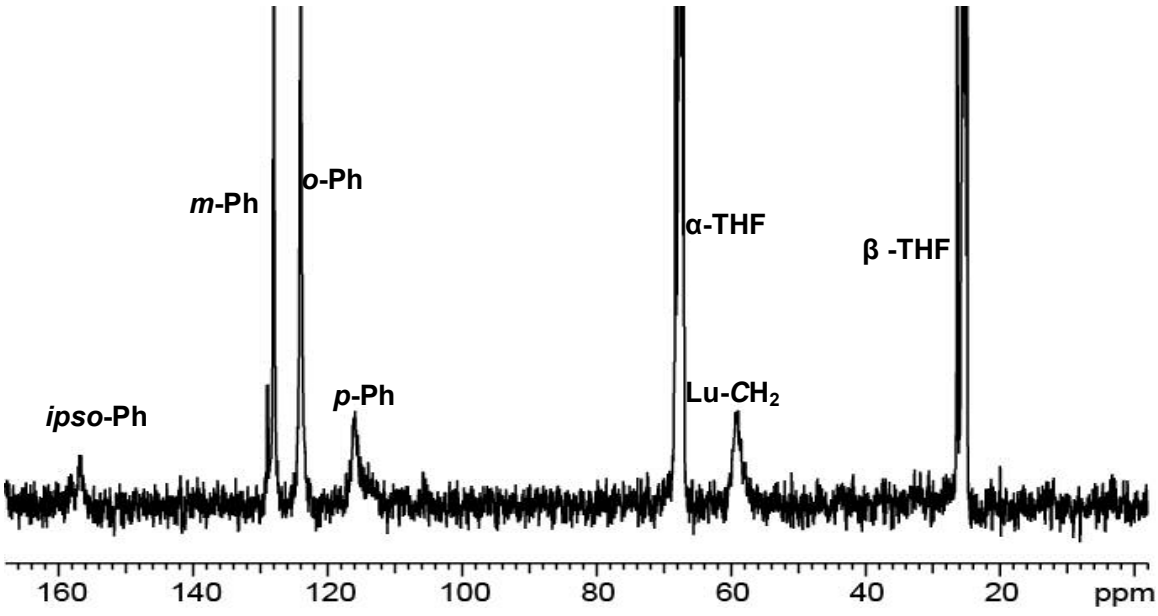


Figure S-16. ^{13}C NMR spectrum (100.58 MHz, THF-d_8, 27°C) of $Lu(CH_2Ph)_3(THF)_3$ (**4**).

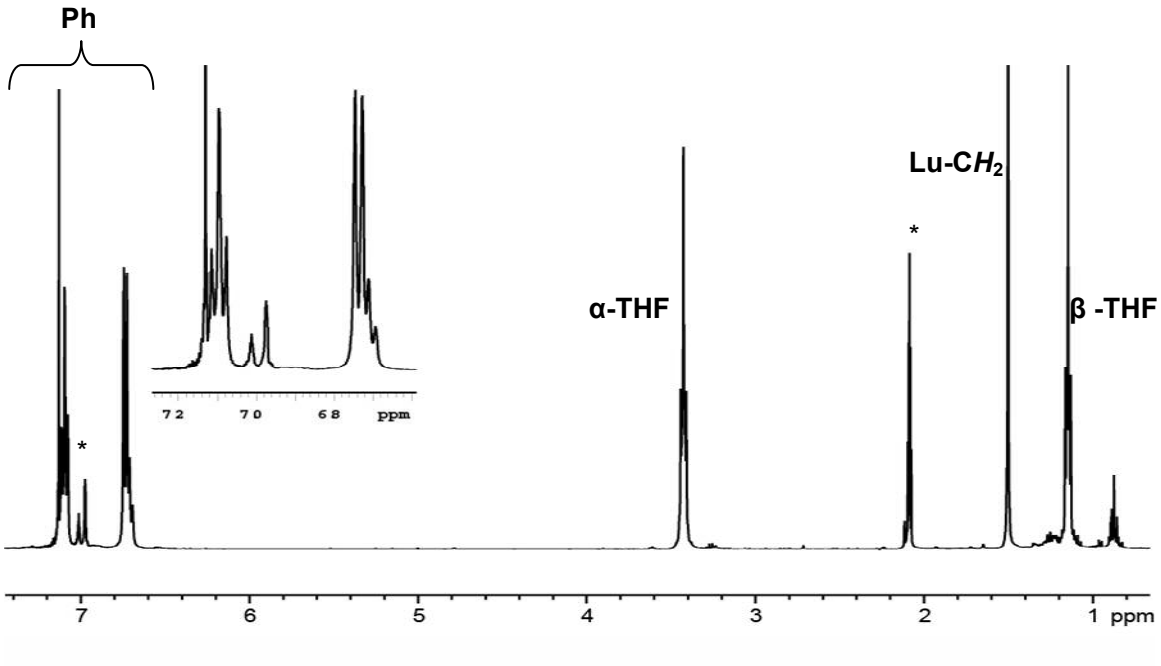


Figure S-17. ^{1}H NMR spectrum (400 MHz, Toluene-d_8, 27°C) of $Lu(CH_2Ph)_3(THF)_2$ (**5**). * denotes residual solvent signals.

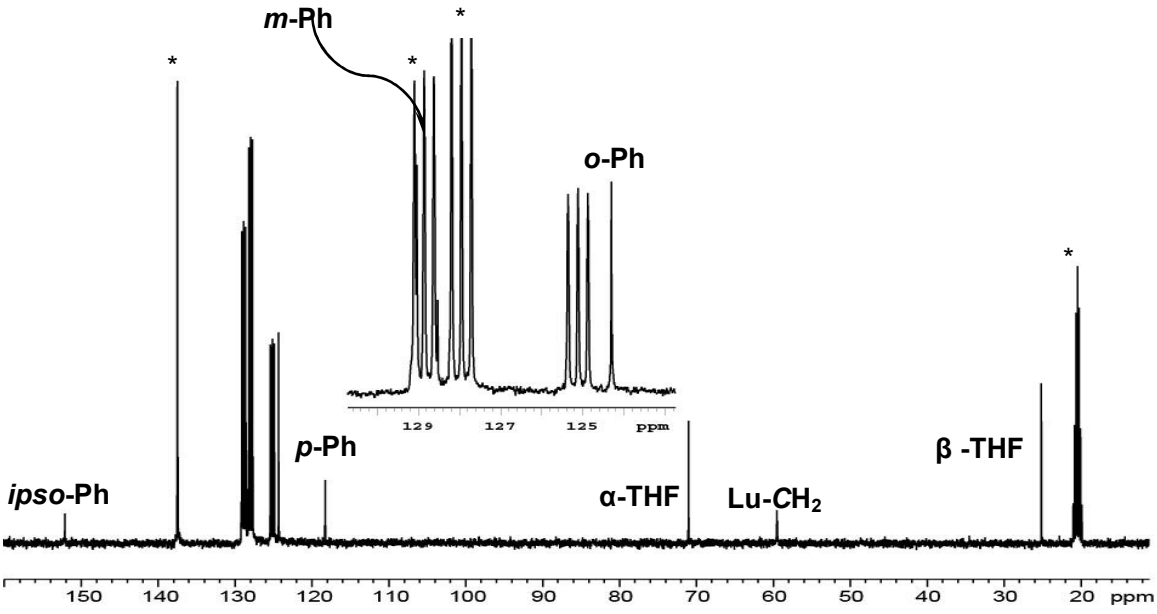


Figure S-18. ^{13}C NMR spectrum (100.58 MHz, Toluene-d_8, 27°C) of $Lu(CH_2Ph)_3(THF)_2$ (**5**). *denotes residual solvent signals.